

# **Field Analytical Measurement Technologies, Applications, and Selection**

***CMECC***

**California Military Environmental Coordination Committee  
Chemical Data Quality / Cost Reduction Process Action Team**

**April 1996**

## Preface

On December 5, 1991, Governor Pete Wilson signed Executive Order W-21-91 to establish the California Base Closure Environmental Committee (CBCEC). The CBCEC's mission was to expedite environmental restoration and reuse of closing military bases in California. In January 1995, the Governor redesignated the CBCEC as the California Military Environmental Coordination Committee (CMECC) by Executive Order W-116-95 and expanded its role to include former and active bases and to also address compliance issues. Member organizations include the U.S. Department of Defense, Military Services (Army, Navy, and Air Force), U.S. Environmental Protection Agency Region IX, and the California Environmental Protection Agency's (Cal/EPA) Department of Toxic Substances Control and State Water Resources Control Board. The CMECC assists both the Governor's Office of Planning and Research and the Office of the Deputy Under Secretary of Defense for Environmental Security via improved interagency communications and cooperative efforts (such as process action teams) that promote cost effective compliance with Federal and State environmental laws and regulations. For example, based on options identified in the CBCEC's December 1994 report, "Options for Cost Reductions in Environmental Assessment", the CMECC determined that significant cost savings may be achieved by the implementation of a guidance document on applications of field analytical measurement technologies. Field measurement technologies can be as sophisticated as fixed laboratory technologies and can generate high quality data faster at reduced costs. Consequently, in June 1995, the CMECC established the Chemical Data Quality/Cost Reduction Process Action Team (PAT) to develop such a guidance document. As directed, the PAT developed this document, ***"Field Analytical Measurement Technologies, Applications, and Selection"***. The information is intended to assist military and regulatory remedial project managers and military base consultants in selecting and applying field analytical measurement technologies. Because this technology is evolving quickly, the PAT anticipates the need to expand and update this document.

## Acknowledgements

This document was prepared through collaborative efforts by all members of the Chemical Data Quality/Cost Reduction Process Action Team (PAT), under the direction of the CMECC. The PAT Chair and point of contact for this document, including the Questionnaire (Appendix E), is Alan Hurt (619/532-3964). PAT members include:

Nars Ancog	U.S. Navy, Southwest Division
Major Eric Banks	U.S. Air Force, Center for Environmental Excellence
Charles Carter	Lockheed/International Association of Environmental Testing Laboratories
David Chia	U.S. Army, Corps of Engineers
Vincent Del Greco	U.S. Army, Corps of Engineers
Tom Flor	U.S. Navy, Facilities Engineering Service Center
Norm Goldstein	Lawrence Berkeley National Laboratory
Robert Haas *	Cal/EPA, Department of Toxic Substances Control
Lisa Hanusiak *	U.S. Environmental Protection Agency, Region IX
Alan Hurt	U.S. Navy, Southwest Division
Mary Obland	U.S. Navy, Engineering Field Activity West
Chuck Owens *	Cal/EPA, State Water Resources Control Board
William Ray	Cal/EPA, State Water Resources Control Board
Roseanne Sakamoto *	U.S. Environmental Protection Agency, Region IX
Barton Simmons *	Cal/EPA, Department of Toxic Substances Control
Major Dan Welch	U.S. Air Force, Center for Environmental Excellence
Peter Wood	Cal/EPA, Department of Toxic Substances Control

The PAT wishes to thank all those who contributed to the development of this document. Asterisks identify the PAT members that participated on the writing subcommittee. PAT meetings were facilitated by Stacey Lupton, PRC Environmental Management. The State Water Resources Control Board printed this document. For copies, call 916/227-4368.

# Contents

	<u>Page</u>
1. Introduction	1
2. Successful Applications of Field Analytical Measurement Technologies	2
3. Field Analytical Measurement Technology Applications in the CERCLA Process	3
3.1 Preliminary Assessment and Site Investigation Phases	3
3.2 Remedial Investigation Phase	4
3.3 Remedial Action Phase	4
3.4 Risk Assessment and Other Phases	4
4. Selection of Field Analytical Measurement Technologies	5
4.1 Field Analytical Measurement Technologies Matrix	5
4.2 Field Analytical Measurement Parameters	6
4.3 Field Analytical Measurement Technologies in the Regulatory Process	7
4.4 Quality of Data Generated by Field Analytical Measurement Technologies	7
4.5 DQOs and Selection of Field Analytical Measurement Technologies	9
5. Establishing Data Quality Objectives for Field Analytical Measurements	9
5.1 The End Goal and DQOs	10
5.2 Field Analytical Measurements and Sampling and Analysis Plans	10
5.3 Revision of Existing SAPs for Field Analytical Measurements	11
6. Field Analytical Measurement QA/QC Program	11
6.1 Initial Field Analytical Measurement Instrument Calibration	12
6.2 Action Level vs. Analytical Measurement Range	12
6.3 Field Analytical Measurement QC Procedures	13
6.4 Laboratory Confirmation	13

6.5 Post-Sampling Analytical Data Review and Validation	15
7. References	16
Appendices:	
A. Field Analytical Measurement Technologies Matrix	Yellow
B. CERCLA, RCRA, UST, and CWA Process Flow Charts	Green
C. Planning Steps for DQO Development/Mare Island NSY Example	White
D. Explanation of Acronyms/Terms	Blue
E. Questionnaire	White

# **Field Analytical Measurement Technologies, Applications, and Selection**

## **1. Introduction**

The evaluation of new field analytical measurement technologies in the U.S. Environmental Protection Agency's (USEPA's) Superfund Innovative Technologies Evaluation (SITE) Program and successful applications at military and private sites has demonstrated that some are capable of generating high quality data at reduced costs and in less time than conventional methods. The goal of this document is to promote cost and time savings by reducing the need for laboratory analyses and remobilization of equipment and contractors through the application of field measurement technologies at former, active and closing military bases. Several available field analytical measurement technologies are presented, their capabilities and limitations, costs, and some possible applications. Sample collection methods are not addressed. Although intended generically, focus is placed on immunoassay, X-Ray Fluorescence (XRF), and the Site Characterization and Analysis Penetrometer System with Laser Induced Fluorescence (SCAPS-LIF).

The Secretary of Defense is required to carry out the Defense Environmental Restoration Program "subject to, and in a manner consistent with", the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) for National Priority List (NPL) sites and non-NPL sites. Therefore, possible applications are identified under the phases of the CERCLA process. The Military Services also follow the Corrective Action process under "RCRA" Subtitle I, or the State process, for underground storage tank (UST) releases which also have opportunities to apply field analytical measurement technologies. Since fuel releases are very common at military bases and several technologies presented are designed to detect petroleum hydrocarbons, significant cost and time savings are possible in UST applications. Document users are encouraged to select field analytical measurement technologies appropriate for their sites in consideration of the technologies' capabilities and limitations consistent with project data quality needs.

The instant availability of field measurement data gives remedial project managers (RPMs) the flexibility to devise more cost efficient sampling and analysis strategies. For example, sites may be fully characterized by analyzing more samples at less cost, or additional sites may be sampled that would have involved subsequent contractor and equipment remobilization. Since the added flexibility may require more initial planning, this document addresses the development of data quality objectives (DQOs) for field measurements in Sampling and Analysis Plans (SAPs). Given the importance of accurate, reliable and defensible data, quality assurance/quality control (QA/QC) for field measurements is also addressed.

## **2. Successful Applications of Field Analytical Measurement Technologies**

Some of the successful applications of field measurement technologies at military bases are presented for RPMs to consider because they may be cost effective in similar applications at other military bases:

Vandenberg Air Force Base. The Central Coast Regional Water Quality Control Board has accepted data generated using immunoassays for total petroleum hydrocarbon (TPH) screening because of demonstrated good correlation with laboratory data and overall cost effectiveness. [The point of contact is Carol Kolb, RWQCB-3, 805/542-5323.]

Treasure Island Naval Station. Immunoassays for polynuclear aromatic hydrocarbons (PAHs) were used for field screening to better locate soil borings and monitor wells. The USEPA's Contract Laboratory Program protocol for semivolatile organic compounds was used. Of the laboratory confirmation sample results, 94% were consistent with field analytical measurements, with 3% false negatives and 3% false positives. This correlation and cost effectiveness resulted in acceptance of the field analytical measurement data by the San Francisco Regional Water Quality Control Board. [The point of contact is Gina Kathuria, RWQCB-2, 510/286-4267.]

Presidio of San Francisco. Immunoassays were used to characterize leaks from USTs and fuel lines in conjunction with 100% laboratory confirmation. Satisfactory correlation with laboratory data resulted in acceptance of the immunoassay data by the San Francisco Bay Regional Water Quality Control Board. [The point of contact is Richard Hiatt, RWQCB-2, 510/286-0699.]

Port Hueneme Naval Construction Battalion Center. The Navy demonstrated that its SCAPS-LIF is capable of rapidly assessing the spatial extent of fuel plumes by cone penetrometer with in-situ qualitative screening for TPH. In one application, 85% accuracy of qualitative results (detect vs. non-detect) was achieved. [The point of contact is Steve Lieberman, NRAD, 619/553-2778.]

Mare Island Naval Shipyard. XRF analyses were performed using an on-site laboratory to determine the extent of metals contamination and ambient concentrations of metals in soils. Key elements of the investigation included: (a) defining project-specific DQOs, (b) implementing sufficiently rigorous analytical quality control procedures, (c) performing confirmation analyses on approximately 15% of the samples, and (d) conducting a data quality assessment. These elements ensured that XRF data suitable for use in the risk assessment for the site were generated for antimony, cadmium, copper, iron, lead, manganese, molybdenum, nickel, silver, and zinc. See Appendix C for DQOs. [The point of contact is Tom Huetteman, USEPA-9, 415/744-2407.]

Sierra Army Depot. Immunoassay test kits were applied to determine the extent of trinitrotoluene 1, 3, 5-hexahydro-triazine (TNT/RDX) contamination in soils. For verification, all samples were also analyzed using USEPA Method 8330, high performance liquid chromatography, then field and laboratory results were compared. The results produced less than 5% false negatives and 0 - 10% false positives. [The point of contact is Robert Haas, DTSC-HML, 510/540-2803.]

North Island Naval Air Station. Immunoassays were applied to detect and delineate polychlorinated biphenyl (PCB) "hot spots" using a grid data point system in an area where transformers were known to have been stored. Good correlation with laboratory data and cost effectiveness resulted in data acceptance by the San Diego Regional Water Quality Control Board. [The point of contact is Charles Cheng, RWQCB-9, 619/467-2975].

### **3. Field Analytical Measurement Technology Applications in the CERCLA Process**

The need to collect and analyze samples is a recurring requirement in the CERCLA site restoration process. The need arises immediately after site discovery to develop the Hazard Ranking Score and continues periodically through to the Remedial Action (RA) phase. Opportunities for significant cost savings exist by analyzing soil and ground water samples using in-situ and ex-situ field analytical measurement technologies to identify, assess, and monitor contaminant sites, particularly, during the Site Inspection (SI), Remedial Investigation (RI), RA phases, and some removal actions. For closing

bases, field measurement technologies also have application in “CERFA” compliance including the identification of “uncontaminated” parcels for reuse and preparation of Environmental Baseline Surveys.

### **3.1 Preliminary Assessment and Site Inspection (PA/SI) Phases**

The PA/SI process is used to determine the severity of contamination for site identification. The purpose of a PA is to: (1) describe the source and key characteristics of a release, (2) characterize the threat posed to human health and the environment, and (3) determine the need for: (a) removal action, or (b) SI, or (c) RI/FS, or (d) no further action (NFA). Field measurement technologies can be cost effective in all of these applications. For example, field measurement technologies may be applied in characterizing the type and source of contamination during the PA to determine the need for a removal action, or SI, or RI. For SI or RA NFA determinations, definitive numeric analytical data are necessary to demonstrate regulatory compliance, so field measurement technologies selected should have this capability. Regulatory acceptance of the field analytical data is essential, so joint planning is strongly recommended.

### **3.2 Remedial Investigation (RI) Phase**

The purpose of a RI is to identify the source and assess the spatial extent of contamination (for remedial engineering design), pathways of migration, and human/ecological impacts. Field investigations conducted during the site characterization portion of the RI include taking in-situ measurements or collecting samples for field and laboratory analyses. It is important that the sampling be phased so that the results from initial sampling can be used to focus future activities including subsequent sampling. Field analytical measurement technologies should be considered for assessing the spatial extent of contamination in soils and ground water, after the CoCs have been determined. Definitive numeric data are necessary to develop isoconcentration contour maps delineating contaminant plumes in ground water or “hot spots” on soils maps, so field analytical measurement technologies used to generate this data must have this capability.

### **3.3 Remedial Action (RA) Phase**

Possible applications of field analytical measurement technologies in the RA phase include monitoring soil treatment effectiveness and long-term ground water monitoring to measure cleanup progress by active remediation methods and natural attenuation of petroleum hydrocarbons. Definitive numeric data are normally necessary to determine compliance with the applicable cleanup levels. Therefore, field analytical measurement technologies for these applications should be selected that have the ability to generate screening or definitive quality data sufficient to satisfy project data quality needs.

### **3.4 Risk Assessment and Other Phases**

The baseline risk assessment, done concurrently with the RI phase, requires definitive numeric analytical data to calculate the risk to human health by exposure to CoCs (carcinogens and non-carcinogens) in the absence of any remedial actions. Field analytical measurement technologies can be used to optimize the strategy for obtaining site-specific fate and transport samples. However, existing laboratory analytical data may already be available for this purpose from earlier SI sampling. Field analytical measurement technologies have few applications in the other phases of the CERCLA process; exceptions include some removal actions and possibly bench scale pilot testing during the Feasibility Study phase.



## 4. Selection of Field Analytical Measurement Technologies

To select an appropriate field analytical measurement technology, RPMs should consider: (a) the capabilities, limitations, and costs associated with the available instruments; (b) the type of release, parameters to be measured in the field, and detection capabilities of the available instruments; (c) the quality of data generated by the available instruments; and (d) applications in the relevant regulatory process and project DQOs. Regulatory RPMs should be involved in the early planning to ensure acceptance of field analytical measurement data. Pilot demonstration tests may well be needed to develop confidence and to calibrate field and laboratory results.

### 4.1 Field Analytical Measurement Technologies Matrix

Appendix A, *Field Analytical Measurement Technologies Matrix*, was designed to assist document users in the selection of field analytical measurement technologies for certain parameters in soil and water samples. The matrix summarizes the capabilities and limitations of several recently developed field analytical measurement technologies. Specifically, detection limits, false negative/positive rates, unsuitable physical conditions, chemical interferences, and costs are provided. The information presented in Appendix A may be used to match field analytical measurement technologies with site-specific conditions and project data needs. The Appendix A matrix is not a complete compilation of currently available field analytical measurement technologies, nor is the information regarding each one comprehensive. Most of these field analytical measurement technologies have been evaluated in the USEPA's SITE Program; they were included in the matrix based on information provided by the manufacturers. Inclusion does not imply endorsement by the CMECC or PAT, nor does exclusion imply negative evaluation. Other field analytical measurement technologies should be considered in addition to those presented in Appendix A. Contact the manufacturers for the latest literature and specifications on technologies in Appendix A. Also, verification of the manufacturers' claims is important when considering staff training needs. The manufacturers' documentation should be consulted for operating procedures.

Although not included in the Appendix A matrix, soil gas surveys and mobile laboratories are recommended because they have proven to be highly cost effective. Soil gas surveys measure the concentrations or flux of volatile organic compounds (VOCs) in soil pores. Both the active and passive methods (for example, Gore-Sorber) are effective in identifying VOCs and assessing the extent of contamination including "hot spots". Active methods involve withdrawing samples of soil gas through a probe driven into vadose soils at shallow depths. Passive methods employ a sorbent sampling device that is buried for a specified time interval, then retrieved for analyses. Samples are commonly analyzed using mobile laboratories to get real time data, usually by gas chromatograph/mass spectrometry (GC/MS). Beyond soil gas surveys, the efficient use of mobile laboratories can greatly accelerate site investigations and the associated decision-making process for the restoration of VOC and non-VOC-contaminated sites. Costs may be less than fixed laboratory analyses depending on the applications and project data needs.

### 4.2 Field Analytical Measurement Parameters

The field analytical measurement technologies presented in Appendix A are designed to detect TPH, PAHs, BTEX, TNT/RDX, PCBs, mercury and other metals. RPMs must identify the constituents of concern (CoCs) at their sites to select field analytical measurement technologies having the appropriate detection capabilities. PA reports normally include readily available information on past military

operations involving chemicals, waste management, and results of past sampling activities, but generally do not include any new analytical data. PAs/records on chemical use operations and waste disposal should be evaluated to identify CoCs. For sites having no historical records, as is often the case for formerly used defense sites (FUDS), laboratory analyses for a broad range of chemicals is often necessary to determine the CoCs. During RI site characterization, analytical testing should focus on contaminants that: (a) are mobile and most likely to reach ground water, (b) provide the best indicators of contaminant migration, (c) pose the highest risk to receptors, and (d) affect remedy selection. Consequently, field analytical measurement technologies should be selected that have the appropriate detection capabilities and can generate the quality of data required for the project under site-specific conditions.

Military bases often use a broad range of petroleum products including lower molecular weight hydrocarbons which are characteristic of stoddard solvent, jet fuels and kerosene as well as heavier diesel fuels, bunker fuel oil and viscous petroleum-based lubricants. It is important to note that no single analytical test method can detect the entire spectrum of petroleum products since none have sufficient sensitivity for all petroleum hydrocarbon compounds. For example, investigation for BTEX in soil using a TPH method may yield a false negative result which limits the applications. Review of the data on the TPH immunoassay test presented in Appendix A is valuable in deciding whether to use the test. Appendix A states: (a) the detection limits are rather high, especially the Ensysis instrument (10 - 40 ppm for soils), (b) the false positive rate is 0 - 45%, (c) an unsuitable condition is water content greater than 30%, and (d) an additional technical comment is that false negative and positive rates determined at 200% and 20% of action level (10 ppm), respectively. Since regulators are unlikely to embrace the test data for NFA and most other determinations given these deficiencies, another selection should probably be considered.

Physically or chemically unstable analytes and certain other parameters should be measured in the field as soon as practicable; examples include pH, redox potential, chlorine, dissolved oxygen, temperature, turbidity, and specific conductance. Most conductivity instruments require temperature compensation. Therefore, the temperature of samples should be measured at the time conductivity is determined unless the instrument automatically makes this compensation. Two methods are generally used for measuring unstable parameters. One method is to use specially designed meters which have probes that may be lowered down well casings or placed in the discharge line from a pump; for example, pH, specific ion electrode, and thermistor. The other method is to collect samples, then analyze them using field measurement instruments. Instruments to measure unstable analytes are not further discussed herein because they have been available and utilized for several years.

### **4.3 Field Analytical Measurement Technologies in the Regulatory Process**

Since the Military Services are subject to other laws in addition to CERCLA, there are four regulatory processes followed nationwide, all of which involve opportunities to apply field analytical measurement technologies. Appendix B consists of flow charts of the four processes which include CERCLA, "RCRA" Corrective Action under Subtitles C (hazardous waste) and I (USTs), and the "Clean Water Act" (CWA) regulatory structure. RPMs should review these flow charts with respect to each site and operable unit at their military bases to identify opportunities to apply field measurement technologies. Several applications in the CERCLA process are identified in Section 3. Several field analytical measurement technologies in Appendix A are capable of detecting petroleum hydrocarbons, common at former, active and closing military bases, and can be applied in all phases of investigation and monitoring in the UST Corrective Action process. To a much lesser degree, field analytical

measurement technologies may be applied in the Corrective Action process for hazardous waste sites under “RCRA” Subtitle C and the CWA regulatory structure (for example, monitoring treated effluent concentrations prior to discharge).

#### 4.4 Quality of Data Generated by Field Analytical Measurement Technologies

Some field analytical measurement technologies are only capable of generating qualitative screening detection results at high concentrations rather than definitive numerical data. However, screening data are sufficient for some applications in the site restoration process. For example, the SCAPS-LIF may be applied to assess the vertical and horizontal extent of fuel plumes which may only require detect/non-detect results to delineate the limits of contamination/clean areas. Whereas, monitoring the effectiveness of a treatment process, or the lack of effectiveness, would normally require definitive numeric data. Table 4.1 identifies the quality of data generated by some currently available field measurement technologies. Understanding the quality of data needed for a specific project is fundamental to selecting the appropriate field analytical measurement technology.

**Table 4.1 Field Measurement Technologies and Quality of Data Generated**

TECHNOLOGY	SCREENING DATA	DEFINITIVE DATA
Immunoassay/Colorimetric	X	X
X-Ray Fluorescence (XRF)*	X	X
Site Characterization and Analysis Penetrometer Systems (SCAPS)	X	
Field Portable Gas Chromatography/Mass Spectrometry (GC/MS)	X	X

Notes: 1. This data is only current as of March, 1996. If necessary, verification reports should be reviewed to verify the type of data that can be generated by a specific technology.

\* 2. XRF data may be considered definitive on a project-specific basis if confirmation data demonstrates acceptable agreement.

For clarity, the USEPA’s definitions of “screening data” and “definitive data” are provided (USEPA 1993):

Screening Data. Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent instead of elaborate extraction/digestion. Screening data provide analyte identification and quantification, although the quantification may be relatively imprecise. At least 10% of the screening data are confirmed using analytical methods and QA/QC procedures and criteria associated with definitive data. Screening data without associated confirmation data are not considered to be of known quality.

Definitive Data. Definitive data are generated using rigorous analytical methods such as approved USEPA reference methods. Data are analyte-specific with confirmation of analyte identity and

concentration. Definitive methods generate tangible raw data (for example, chromatograms, spectra, digital values) in the form of paper printouts or computer generated electronic files. Data may be generated at the site or at a fixed laboratory provided the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined.

#### **4.5 The DQO Process and Selection of Field Analytical Measurement Technologies**

DQOs are quantitative and qualitative specifications applicable to field data collection. The seven planning steps for DQO development, known as the DQO process, are listed in Appendix C with an example. Selection of field analytical measurement technologies should be based on the outputs of the first six steps of the DQO process. The type, quality, and quantity of samples planned for collection must be checked to ensure they meet project data needs. In the first six steps, RPMs develop the DQOs that will be used to develop the data collection strategy. The first six steps should be completed before RPMs attempt to develop the data collection strategy because Step 7, which is the final step, is dependent on a clear understanding of the first six steps taken as a whole. Step 7 involves developing the data collection strategy and selection of the field analytical measurement technology based on the DQOs. This final step is used to determine if field analytical measurement technologies will actually perform as needed to meet the project data quality requirements (for example, to determine if detection limits are sufficient). All seven steps should be completed before field data collection begins.

### **5. Establishing Data Quality Objectives for Field Analytical Measurements**

The ability of field analytical measurement technologies to generate analytical data for multiple phases of the CERCLA process and to make decisions in the field empowers RPMs to consider basewide sampling strategies which requires more up-front planning. Planning includes the development of DQOs for field analytical measurements. Project-specific DQOs are generally based on an action level, for example, background (unaffected by waste release) or Water Quality Objectives. Because of the importance of accurate, reliable, and defensible analytical data to the site restoration process, DQOs should be established early in the project and incorporated into the SAP. DQOs should be developed prior to the first field data collection effort and be applied to subsequent data collection activities. DQOs should be re-examined periodically (for example, at each point in the project when additional data are required) to ensure that field analytical measurement data quality continues to meet project data needs.

#### **5.1 The End Goal and DQOs**

RPMs should establish DQOs with the end goal of the project in mind and develop cost effective strategies for data collection. This includes maximizing cost and time savings by applying field analytical measurement technologies. The end goal should be inclusive of the different phases of investigation in the applicable regulatory process. When DQOs are established with the immediate objectives and end goal in mind, the data may be used for multiple purposes. For example, if the final cleanup goal could be one of two different action levels, the more stringent action level should be used as a guide for the detection limit requirement since it will also satisfy the less stringent action level. Establishing DQOs that can meet the data needs of multiple phases of the CERCLA process can avoid unnecessary equipment remobilization and reduces laboratory costs. There are times when a single phase approach may be necessary. However, the availability and sophistication of field analytical measurement technologies no longer restrict investigations to a single phase approach. The success of any sample collection activity is dependent on knowing the quality of the data needed and making sure

that the data is generated in a manner consistent with the intended use. The DQO process is for developing a scientific and resource-effective sampling strategy; see Appendix C for the DQO process.

## 5.2 Field Analytical Measurements and Sampling and Analysis Plans

The SAP is developed during the scoping phase of the RI/FS. A SAP is composed of two parts, the Field Sampling Plan (FSP) and the Quality Assurance Project Plan (QAPP). The FSP identifies the methods to be used for field sampling and data gathering activities while the QAPP identifies the DQOs. Although the FSP is prepared before commencement of RI field work, the SAP should be re-evaluated and revised as the requirements and scope of field work evolve during the site restoration process. However, contingencies for the collection of additional samples, dependent on the results of SI sampling, should be anticipated and proposed in the SAP to avoid unnecessary SAP revisions. SAPs should be based on decision end points and performance criteria. For example, if the decision is NFA at a soils site or to conduct further testing, the criteria should be stated, e.g., Preliminary Remediation Goals for the CoCs.

The key elements of the FSP include:

Objective	Disposal of contaminated materials
Background	Equipment decontamination
Maps	Sample containers
Sample locations, number & parameters	Sample preservation
Request for analyses	Sample packaging and shipment
Field methods and procedures	Sample documentation
Sample collection	QC sampling

The key elements of the QAPP include:

Project description	Data reduction, validation, reporting
Project organization and responsibilities	Internal QC procedures
QA objectives for measurement	Performance and system audits
Sampling procedures	Preventive maintenance
Sample custody procedures	Data assessment procedures
Calibration procedures	Corrective actions
Analytical procedures	QA reports

The QAPP should list the specific parameters to be measured in the field. It should specify the type of instruments that will make the measurements and describe the procedures to be followed in operating the instruments and recording measurements. The QAPP should specify QC objectives for precision, accuracy, bias, completeness, representativeness, and comparability for both field and laboratory analyses that will ensure that project DQOs are met. The QAPP should describe all instrument calibration procedures including: (a) the frequency of calibration, (b) discussion of initial calibration, (c) determination of usable range, (d) periodic calibration checks, (e) conditions that warrant re-calibration, (f) acceptable control limits, and (g) the maintenance of calibration records in a field log book. All field analytical instruments should be calibrated using a procedure appropriate for the site-specific application. For example, field screening tests should include a calibration standard appropriate for the action level used. For more information on QAPP content, please refer to "EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations", EPA QA/R-5, Draft, January 19, 1992.

### **5.3 Revision of Existing SAPs for Field Analytical Measurements**

Most active and all closing military bases in California already have approved SAPs. It may be appropriate to revise SAPs to incorporate field analytical measurements depending on remaining project data needs and the potential for cost savings. In addition to SAP revision, Health and Safety Plans may need to be revised for field measurement applications involving possible exposure to hazardous chemicals.

## **6. Field Analytical Measurement Quality Assurance/Control Program**

One of the base RPM's responsibilities is the establishment of a continuing QA/QC program to ensure the reliability and validity of field and laboratory analytical data. An experienced chemist should be involved in establishing QC procedures for field measurements and selecting a reliable fixed laboratory to perform confirmation analyses. The QAPP should provide for the collection and analyses of QC samples including training for the staff that make field analytical measurements. Various types of QC samples and blanks should be used to verify that the sample collection and handling process has not affected the quality of the samples. Recommended QC sampling frequency for field analytical measurements are presented in Table 6.1. Blanks and QC samples should be prepared and analyzed using the same procedures as those used for environmental samples. The QC samples should be analyzed for all of the required monitoring parameters. Both field and laboratory QC samples should be prepared during the sampling event. To minimize cross-contamination, sample extraction should be done in an area physically separate from the area used for the testing of extracts.

### **6.1 Preliminary Field Calibration**

A preliminary site-specific calibration check of field analytical measurement instruments should be performed by analyzing split samples; split samples should be analyzed using the field instrument and a reference control method at a fixed laboratory. If there is a significant difference between field analytical measurement and reference method results, a site-specific conversion factor may possibly be developed to adjust the field results provided there is acceptable correlation between the two sets of results and regulatory acceptance. For example, some PCB immunoassays are calibrated with Aroclor 1254; so if PCB contamination is actually due to Aroclor 1242, the response could be 21% of the actual concentration. Cross-reactivities for common contaminants are listed in certification documentation and in the manufacturers' documentation. In some cases, indicator measurements may be used as a

surrogate for a CoC. For example, TPH might be used as an indicator for lower molecular weight (e.g., gasoline) PAH contamination at the same site, but only if a correlation between TPH and PAHs is established for that site. If a site-specific conversion factor can be established for a field analytical measurement instrument that generates only qualitative screening results (for example, PCB results), the accuracy of the conversion factor must be evaluated in comparison with the reference method results.

## 6.2 Action Level vs. Measurement Range

Instrumentation having the highest accuracy should be used to measure analyte concentrations equivalent to the action level. For example, an immunoassay is most accurate at the middle of its measurement range. Therefore, if a target level of 5 ppm PCB in soil was applied with a test kit having a detection range of 1 to 10 ppm, a result of ppm would classify a sample concentration as being below the target level, whereas, a result 10 ppm would classify the sample concentration as being above the target level. Samples with analyte concentrations of 1 ppm to 10 ppm may be sent to a fixed laboratory for definitive analysis using a quantitative reference method. Method detection limits should be at least one half of the applicable action levels. Appendix A includes detection limit data for field analytical technologies.

## 6.3 Field Analytical Measurement QC Procedures

Project-specific DQOs generally determine the specific QC procedures needed. Recommended QC sample frequency for field analytical measurements are presented in Table 6.1. The required frequency of laboratory confirmation depends on the application and project-specific DQOs. For example, if screening is being performed to confirm that a soils site is clean, all positive results should be confirmed using a definitive analytical technique. Definitive confirmation entails that some fraction of the screening data is confirmed with definitive data generated using an independent, accepted method. As a minimum, at least three screening samples reported above the action level (if any) and three screening samples below the action level (or as non-detects) should be randomly selected from the appropriate group and confirmed. Confirmation is discussed in further detail in Section 6.4.

**Table 6.1 Recommended Quality Control Sample Frequency for Field Analytical Measurements**

Quality Control Samples	PA and SI Samples	RI and RA Samples
Method blanks	5% or 1/batch or 1/matrix, whichever is most frequent	5% or 1/batch or 1/matrix, whichever is most frequent
Field duplicates	5% or 1/batch or 1/matrix, whichever is most frequent	5% or 1/batch or 1/matrix, whichever is most frequent
Performance Evaluation (PE) samples or spike samples	Project-specific	Site-specific DQOs
<u>Confirmation</u> field result action level (AL)	10% (1 minimum)	Site-specific DQOs
detection limit (DL) field result AL	10 to 20%	Site-specific DQOs
Field result DL	10%	Site-specific DQOs

(Note: A batch is the set of samples analyzed by the same staff in one day; usually 20 or fewer samples.)

#### 6.4 Laboratory Confirmation

Confirmation is the verification of the results obtained by the field analytical measurements and is performed by comparing the field measurement results with those by laboratory methods. For this reason, the laboratory selected to perform confirmation analyses must have demonstrated proficiency in performing the method in order for the comparison and confirmation to be valid. Initially, a higher percentage of field measurement results should be confirmed with laboratory results. To ensure good agreement between field and laboratory data and for increased confidence in using the field measurement data, confirmation analyses should be performed to cover the range of concentrations expected at the site. After confidence that field and laboratory results are both comparable and consistent, the frequency of confirmation can be reduced to the minimum QC samples stated in Table 6.1 or in accordance with the project-specific DQOs. The recommended frequency of confirmation sampling is presented in Table 6.1 and examples of laboratory confirmation methods in Table 6.2.

**Table 6.2 Recommended Quantitative Laboratory Confirmation Methods**

Field Measurement Parameters	Confirmation Method
TPH	EPA Method 8015A (GC-FID) California LUFT Manual Method
BTEX	EPA Method 8021A (GC-PID/ELCD) or EPA Method 8260 (GC/MS) or CLP Volatile Organics by GC/MS
TNT/RDX	EPA Method 8330 (HPLC)
PAHs	EPA Method 8270B (GC-MS) or CLP Semi-volatile organics by GC/MS or EPA Method 8310 (HPLC)
Mercury in soil	EPA Method 7471A (Cold-Vapor AA)
PCBs	EPA 8080A, 8081A or 8082 (proposed) (GC-ECD or HSD) CLP Chlorinated Pesticides and PCBs
Metals by XRF Spectroscopy (Note: Some partial digestion methods, e.g., 3050A, may give lower results than XRF. The DQOs should determine whether 3050A or the proposed 3052 will be used for confirmation.)	Soil: EPA 3050A (acid digestion) or EPA 3051 (microwave assisted acid digestion) , 3052 (proposed - microwave assisted acid digestion of siliceous and organically based matrices) EPA 6010 (ICP-AES) or 6020 (ICP-MS) CLP Inorganics by ICP-AES
SCAPS (PAH/TPH)	See PAHs methods listed above.



Additional QC procedures should be considered depending on site-specific conditions and project data quality requirements. Whenever feasible, samples should be split in the field. If splitting soil samples is not feasible (for example, for the analysis of VOCs in soil), the splitting of field solvent extracts should be considered in addition to the collection of collocated samples.

## **6.5 Post-Sampling Analytical Data Review and Validation**

Some of the recommended procedures for data review and validation during and after sampling follow:

Review of Field Measurement QC Data. Field analytical measurement data should be reviewed in the field for consistency with the instrument operating procedures, QC sample results and procedures, and project DQOs. Confirmation analyses and re-analyses of samples should be done in the field in accordance with the SAP QA program.

Validation of Laboratory Confirmation Data. All initial data sets obtained from laboratory and field analytical measurements should be reviewed to determine how well the data sets agree. A larger percentage of data should be validated for the initial data sets. At a minimum, 10% of the data generated should be validated according to procedures consistent with those specified in, “USEPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review” (February 1994), as appropriate. Review of a larger percentage of data may be warranted based on project-specific DQOs or if serious analytical problems emerge. Contaminants found in blanks may be due to: (1) interaction between the sample and its container; (2) contaminated rinse water; (3) contaminated preservatives; or (4) the handling procedures used. The concentrations of any contaminants found in blanks should not be used to correct data. Contaminant concentrations should be noted, and if an order of magnitude greater than the field measurement data, the media should be re-sampled and re-analyzed.

Comparison of Field Measurement Data and Laboratory Confirmation Data. Sample homogeneity should be considered in comparing field analytical measurement data and laboratory confirmation data. The false negative and false positive rates listed in Appendix A were obtained using homogeneous samples and may be statistical approximations in some cases. Varying soil conditions and possible interferences encountered may lead to wide variations in false negative and positive rates. These variations may be investigated by using a field trial with a high number of confirmation samples sent to a fixed laboratory for analyses (for example, 50 to 100% of samples). Evaluation of results near the detection limit of any method is problematic. If DQOs for a specific project require measurement near the detection limit, performance evaluation samples or spiked samples should be used as part of QC. Comparison of field measurements and laboratory results should also consider the action levels for the project. Special considerations for XRF and VOCs results follow:

XRF Results. Comparison of X-Ray Fluorescence (XRF) Spectroscopy and laboratory results should consider that XRF is a total metals analysis, but the USEPA digestion methods in Table 6.2 may not completely digest soil samples. Consequently, XRF results will include metals in some form (such as silicates) which are not digested by Method 3050A. Therefore, XRF field measurement results may be consistently higher than laboratory confirmation results.

VOC Results. Samples for VOC analyses (for example, BTEX or TPH) should be preserved if not analyzed within 24 hours. To inhibit biodegradation, water samples must be preserved by acidification with sodium bisulfate or hydrochloric acid prior to shipment for laboratory analysis. Soil samples

should be collected using a single transfer device, then preserved, as appropriate. If soil samples are not preserved, laboratory confirmation results may be lower than field measurements due to volatilization.

Compare Split Sample Results. In general, field screening results and laboratory confirmation results for homogeneous split samples should be within a factor of 10; for example, a field result of 10 - 1,000 ppm should yield a laboratory confirmation of about 100 ppm). XRF results and confirmatory results may be evaluated by regression analysis. A correlation coefficient of 0.9 or greater indicates that the field data may be definitive (USEPA, 1995). In general, if the frequency of false positives or false negatives exceeds the project DQOs, corrections should be made which can involve re-analysis using field and laboratory methods, if holding times allow.

Comparison of Collocated Sample Results. Because of sample heterogeneity, results for collocated samples (collected side-by-side) may differ much more than split sample results, perhaps up to two orders of magnitude. If heterogeneity is suspected, sample extracts can be split in some cases to minimize the effects of sample heterogeneity.

## **7. References**

Ancog, Nars, U.S. Navy Southwest Division.

Anderson, John, San Diego Regional Water Quality Control Board (communication with C. Owens).

Ashley, Kasey, North Coast Regional Water Quality Control Board (comments to C. Owens).

American Society for Testing and Materials, "Guide for Accelerated Site Characterization for Confirmed or Suspected Petroleum Releases" (in preparation).

Cal/EPA, "Documentation for Environmental Technology Certification" (published for each certification).

Cal/EPA, "Interim Final Guidance Manuals for Ground Water Investigations".

Francoeur, Thomas L., "Finding Success With Field Chemistry - The Importance of Defining Data Quality and Project Objectives", Environmental Laboratory, July 1995.

Francoeur, Thomas L., "Field Chemistry - Part II - Defining Project Goals and Objectives", Environmental Testing and Analysis. Volume 4, Number 3, May/June 1995.

Gobler, Eric, Central Coast Regional Water Quality Control Board. (communication with C. Owens)

Haas, Robert, Cal/EPA Department of Toxic Substances Control (lead on matrix preparation).

Hanusiak, Lisa, USEPA-9.

Hoe, Mae, Central Valley Regional Water Quality Control Board (comments to C. Owens).

Huetteman, Tom, USEPA-9 (communication with L. Hanusiak).

Kolb, Carol, Central Coast Regional Water Quality Control Board (communication with C. Owens).

Lee, Shin-Roei, San Francisco Regional Water Quality Control Board (communication with C. Owens).

Lewis, Brian, Cal/EPA Department of Toxic Substances Control (subcommittee meeting).

Lieberman, Steve, U.S. Navy Southwest Division.

LeBlanc, Lindsay M., Cal/EPA State Water Resources Control Board (assistance to B. Ray).

Owens, Charles (Chuck) R., Cal/EPA State Water Resources Control Board.

PRC Environmental Management, Inc., Mare Island Naval Shipyard, Vallejo, California, April 24, 1995.

Ray, William, Cal/EPA State Water Resources Control Board.

Sakamoto, Roseanne, USEPA-9.

Simmons, Barton, Cal/EPA Department of Toxic Substances Control.

Stephens, Bob, Cal/EPA Department of Toxic Substances Control.

U.S. Army Corps of Engineers, Hazardous and Toxic Waste Environmental Laws and Regulations, 1994.

U.S. Department of Energy, “Draft On-Site Measurements; A Guidance Document for Decisions Using On-Site Analytical Support”.

USEPA, “Data Quality Objectives Process for Superfund”, Interim Final, EPA/540/G-93/071, September 1993.

USEPA, “Draft Field Methods Compendium”, OER 9285.2-11, July, 1993.

USEPA, “Field Measurement Techniques: Dependable Data When You Need It”, USGPO 055-000-00368-8.

USEPA, “Field Portable X-Ray Fluorescence Spectrometry for the Determination of Metals Concentrations in Soil and Sediment”, Office of Research and Development, National Exposure Research Laboratory, Characterization Research Division, Las Vegas, Nevada, July 24, 1995.

“USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review”, EPA 540/R-94—012, February 1994.

“USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review”, EPA 540/R-94-013.

USEPA, “Guidance for the Data Quality Objectives Process”, EPA QA/G-4, Final, September 1994.

Ward, Kim, Cal/EPA State Water Resources Control Board (comments to C. Owens).

Youngs, Margie, Cal/EPA State Water Resources Control Board (comments to C. Owens).